MS36 P01

Crystallography with a dual source diffractometer at Reading. Yu Gan[a], R. Jeremy H. Davies[b], Anna. L. Brogden[a], Abeer Naseer[a], Sayima. J. Ahmed[a], Christine. J. Cardin[a]. [a]*Department of Chemistry, University of Reading, RG6 6AD UK.* [b]*School of Biological Sciences, Queen's University, Belfast BT7 1NN UK*
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Keywords: copper source, absolute configuration, DNA

A newly installed diffractometer with facile switching over between copper and molybdenum sources has made the crystallographic studies at Reading more versatile, and this poster demonstrates this with some recent results. The R_{int}s of the data collected with the molybdenum source very often stay at around two percent, which makes the structure solution for difficult structures become possible. On two occasions where Br^- or I^- were included in the structure as counter ions, the absolute configuration of the chiral molecule was determined with very reliable statistics. On the other hand, with a focused copper source also available, the determination of the absolute configuration of typical organic molecules can be routinely carried out. An excellent example is the high resolution crystal structure of the intramolecular thymine-adenine photoadduct d(TpA)* (Figure 1), in which the absolute configuration at the original thymine C5 and C6 atoms confirm the hypothesized photoaddition mode that should be favored by the stacked thymine and adenine bases in B-DNA. All hydrogen atoms were readily located. [1] The stronger copper source also makes it possible to study structures of supramolecular crystals, which very often have tiny crystal sizes. These crystals, as well as being tiny, often have cell dimensions extending to 40-50Å or more, when the long wavelength of copper makes the diffraction spots better resolved. For the same benefits, studies on DNA structures and their interactions with small ligands, and protein crystal engineering utilizing lysozyme and thaumatin as the subjects are also carried out. Nice results like DNA-intercalator complexes, DNA Holliday junctions formed by new sequences, and new A-DNA structures are obtained on a regular basis.

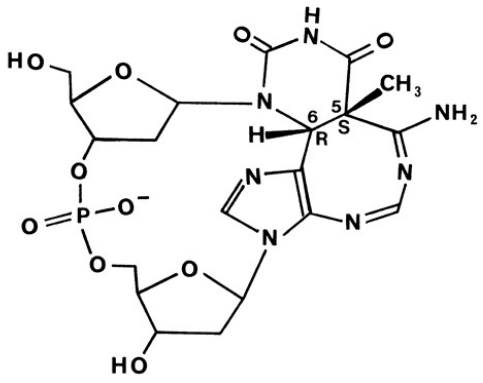

Figure 1 Structure of d(TpA)*

[1] Davies, R. J. H., Malone, J. F., Gan, Y., Cardin, J. C., Lee, M. P. H., Neidle, S. (2006), *Nucleic Acid Res*, **35**, 1048-1053.

MS36 P02

Experiences with a High-Brilliance Microfocus Sealed Tube Juergen Graf, Carsten Michaelsen, Christian Hoffmann, *Incoatec GmbH, Max-Planck-Straße 2, D-21502 Geesthacht.* E-mail: info@incoatec.de

Keywords: X-ray optics, multilayer thin films, new XRD technology

With the gaining importance of macromolecular crystallography came an increasing need for highly intense X-ray sources enabling the analysis of very small and weakly scattering samples. High-brilliance microfocusing X-ray sources are characterized by high power loads in spot sizes of ≤ 100 μm at the anode and deliver an intense but divergent beam which demands for the use of an X-ray optics. Synthetic multilayer mirrors are well established as excellent beam-shaping devices with a very good spectral purity [1, 2, 3, 4]. Their high reflectivity, broad rocking curve and tunable beam divergence and cross-section make them the ideal focusing optics for conserving the source brilliance [5, 6].
New microfocusing sealed tube X-ray sources, such as the *Incoatec Microfocus Source* (IμS^{TM}), are low-maintenance high-brilliant sources which significantly improve the performance of home-lab instruments when combined with dedicated multilayer mirrors. IμS^{TM} contains a 30 W air-cooled microfocus sealed tube with high brilliance and a high-performance 2D Montel multilayer mirror. IμS^{TM} is available for Cu-K_α and Mo-K_α radiation
We will present results on the use of IμS in protein and small molecule crystallography and in small angle X-ray scattering which show that the performance of such an air-cooled microfocusing sealed tube is much better than that of standard sealed tube systems and comparable to traditional rotating anode sources but with a significantly reduced maintenance.

[1] M. Schuster, H. Göbel, *Adv. X-Ray Anal. 39*, 57 (1997).
[2] M. Schuster, H. Göbel, L. Brügemann, D. Bahr, F. Burgäzy, C. Michaelsen, M. Störmer, P. Ricardo, R. Dietsch, T. Holz, H. Mai, *Proc. SPIE 3767*, 183 (1999).
[3] C. Michaelsen, J. Wiesmann, C. Hoffmann, A. Oehr, A. B. Storm, L. J. Seijbel, *Proc. SPIE 5193*, 211 (2003).
[4] A. B. Storm, C. Michaelsen, A. Oehr, C. Hoffmann, *Proc. SPIE 5537*, 177 (2004).
[5] M. Bargheer, N. Zhavoronkov, R. Bruch, H. Legall, H. Stiel, M. Woerner, T. Elsaesser, *Appl. Phys. B 80*, 715 (2005).
[6] J. Graf, C. Michaelsen, J. Wiesmann, A. Oehr, C. Hoffmann, *Acta Cryst. A 62*, s94 (2006).

MS36 P03

A New Flexible High Resolution Powder Neutron Diffractometer at IFE in Norway Klaus Lieutenant[a], Bjørn C. Hauback[a], Helmer Fjellvåg[b], Phillip Bentley[c] [a]*Physics Department, Institute for Energy Technology, Kjeller, Norway.* [b]*University of Oslo, Norway.* [c]*Hahn-Meitner-Institut, Berlin, Germany.*
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Keywords: neutron diffraction; powder diffractometer; Monte Carlo treatment

Various parameters of a neutron powder diffractometer can be varied to increase the flux at the sample. Additionally or alternatively the detector area can be increased to yield a higher count rate. The increased intensity comes on the cost of resolution and precision of

line positions. Here we present a comprehensive study of these effects by means of Monte Carlo simulations followed by data evaluation. Results of an optimization that includes intensity, resolution and line precision are presented. These findings have been used for the design of the new diffractometer ODIN at the JEEP II reactor at IFE in Norway. It will be a flexible, high-resolution instrument. Take-off angle, collimation, monochromator radius and illuminated monochromator area can be varied to adapt the instrument to the needs for the measurement in terms of intensity, resolution and line precision.

MS36 P04

Pseudo-merohedral twinning : how to treat a six-fold twin Leo Straver and Rob Hooft, *Bruker AXS, Oostsingel 209, NL 2612 HL Delft, The Netherlands*
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Keywords: multiple domains, twins, data reduction

The introduction of two dimensional detectors for single crystal diffraction has made it possible to easily collect data of pseodo-merohedral twins, incommensurate structures and structures including diffuse scatter. Many programs exist, e.g. GEMINI [1], CELL_NOW[2] and DIRAX[3], with which these more complex matrices can be indexed. A common problem with pseudo-merohedral twinning is cell refinement due to closely overlapping spots. Careful selection of trusted areas to refine the cell in, will improve the reliability of the cell parameters. Recent versions of integration programs, such as SAINT [4] and EvalCC [5] utilize the different orientation matrices and can integrate the entire intensity of every reflection labeling them as overlapping or not. One of the pitfalls during integration of data from pseudo-merohedral twins is that the spot shape changes continuously due to the systematic overlap of areas of adjacent spots. Special care has to be taken when setting up the integration parameters. Most semi-empirical scaling and absorption correction programs are not able to handle datasets of such complexity. The program TWINABS was developed [6] specifically to carry out absorption correction and scaling on datasets which contain reflections from different domains. Resultant data files distinguish between non-overlapping and overlapping reflections [7] and can be seamlessly used for structure refinement. The approach is illustrated using a six-fold twin that shows a reversible transition to single crystal state.

[1] Sparks, R. (1999). GEMINI, Bruker AXS Inc. Madison.
[2] Duisenberg, A.J.M. (1992). *J. Appl. Cryst.*, **25**, 92-96.
[3] Sheldrick, G.M. (2003). CELL_NOW, Bruker AXS Inc. Madison.
[4] Bruker (2001). SAINT, Bruker AXS Inc. Madison.
[5] Duisenberg, A.J.M., Kroon-Batenburg, L.M.J. & Schreurs, A.M.M. (2003). *J. Appl. Cryst.* (2003), **36**, 220-229.
[6] Sheldrick, G.M. (2002). TWINABS, Buker AXS Inc., Madison.
[7] Sheldrick, G.M. (1997). SHELXL-97, University of Göttingen.

MS36 P05

Non-Conventional Scattering Studies of Materials using a Laboratory Image Plate Diffractometer Lynne H. Thomas[a], Sylvia E. McLain[b], Andrew Parkin[a], and Chick C. Wilson[a]. *a. Department of Chemistry and WestCHEM Research School, University of Glasgow, Glasgow, G12 8QQ, U.K. b. ISIS Facility, Rutherford Appleton Laboratory,* ***Chilton, Didcot, Oxon, OX11 0QX, U.K.***

Keywords: Image plate; diffuse scattering; liquid scattering

Recent trends in the development of X-ray diffractometers have been towards fast readout detectors such as CCDs. Image plate detectors, however, have the advantage that long exposure times are possible due to their high dynamic range, allowing weaker scattering to be observed without suffering from detector overloads or from dark current accumulation. Experiments involving measurements such as liquid scattering and diffuse scattering which have previously required the use of high flux synchrotron radiation have been demonstrated to be possible on a laboratory diffractometer using a curved image plate detector. Diffuse scattering has been observed to arise from a wide range of different materials and its presence is often unexpected. The mechanism behind such scattering is often complex, however insights can be gained from consideration of the average structure of the material and the comparison to similar features in systems where the disorder has been characterised using modeling techniques such as Monte Carlo and Reverse Monte Carlo. The feasibility of collecting liquid diffraction patterns with the aim of studying the early stages of molecular arrangement within solution prior to crystallisation has also been investigated and preliminary results arising from this will be presented.

MS36 P06

The Present and the Future of Protein Microcrystallography at SPring-8 M. Kawamoto[a], N. Shimizu[a], K. Hasegawa[a], A. Nisawa[b], G. Ueno[b], K. Hirata[b], T. Kumasaka[a] and M. Yamamoto[a,b], [a]*SPring-8/JASRI,* [b]*RIKEN SPring-8 Center*
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Keywords: microcrystals, microbeam, synchrotron radiation

BL41XU is an undulator beamline at Japanese third-generation synchrotron facility SPring-8. This beamline has been improved for obtaining high quality data from protein micro crystals (~ 25 μm) using a micro beam (~ 25 μm). The new K/B mirror system was installed at the autumn of 2006, and the monochromatized beam is focused to V70 × H100 μm (F.W.H.M.) at sample position. The final beam size at sample position is defined from 25 × 25 to 70 × 100 μm^2 (F.W.H.M.) by using two set of quadrant slits. Photon flux and flux density of the 25 × 25 μm^2 beam at 1 Å are 3.0×10^{11} photons/sec and 4.8×10^{14} photons/sec/mm^2, respectively.
It is essential for the data collection to control the radiation dose since micro crystals will be received remarkable radiation damage. The data collection software installed to BL41XU can change the beam irradiation position automatically to suppress the effect of radiation damage by using multiple positions on a crystal and multiple crystals. SAD measurements of Se-methionine samples with the crystal size from 15 to 50 μm were performed by using this tool, and we successfully obtain the initial phase at the resolution from 2.7 to 3.9 Å.
A new micro focus beamline in order to utilize the real micro beam; target beam size is 1 × 1 μm^2, is planned at SPring-8. We present the present status of protein